

FORM PTO-1390  
(REV 12-29-99)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

30098.4

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/554733

INTERNATIONAL APPLICATION NO.  
PCT/EP98/07599INTERNATIONAL FILING DATE  
November 25, 1998PRIORITY DATE CLAIMED  
December 3, 1997

TITLE OF INVENTION SPONGE CLOTH BASED ON CELLULOSE AND PRODUCTION THEREOF

APPLICANT(S) FOR DO/EO/US KALLE NALO GMBH &amp; CO. KG

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
  2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
  3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
  4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
  5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
    - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
    - b. ☒ has been transmitted by the International Bureau.
    - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
  6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
  7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
    - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
    - b. ☐ have been transmitted by the International Bureau.
    - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
    - d. ☒ have not been made and will not be made.
  8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
  9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
  10. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).
- Items 11. to 16. below concern document(s) or information included:
11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
  12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
  13. ☒ A **FIRST** preliminary amendment.  
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
  14. ☐ A substitute specification.
  15. ☐ A change of power of attorney and/or address letter.
  16. ☒ Other items or information: Amended Sheets pp. 2, 2a, 8, 9, and 11; forms 409, 416, 210, 308, 332, 304, 301, 105 and 101.

09/554733

PCT/EP98/07599

30098.4

17. ☒ The following fees are submitted:

422 Rec'd PCT/PTO 19 MAY 2000

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):

Neither international preliminary examination fee (37 CFR 1.482)  
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO  
and International Search Report not prepared by the EPO or JPO ..... \$970.00

International preliminary examination fee (37 CFR 1.482) not paid to  
USPTO but International Search Report prepared by the EPO or JPO ..... \$840.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but  
international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$690.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)  
but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$670.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)  
and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$96.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 840.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	20 - 20 =	0	X \$18.00
Independent claims	3 - 3 =	0	X \$78.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$260.00

\$

TOTAL OF ABOVE CALCULATIONS =

\$

Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement  
must also be filed (Note 37 CFR 1.9, 1.27, 1.28).

\$

SUBTOTAL =

\$

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

TOTAL NATIONAL FEE =

\$ 840.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be  
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$ 40.00

TOTAL FEES ENCLOSED =

\$ 880.00

Amount to be  
refunded: \$  
charged: \$

- a. ☐ A check in the amount of \$\_\_\_\_\_ to cover the above fees is enclosed.
- b. ☒ Please charge my Deposit Account No. 023292 in the amount of \$ 880.00 to cover the above fees.  
A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
overpayment to Deposit Account No. 023292. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

**Bowles Rice McDavid Graff & Love, PLLC**  
475 H Street  
Suite 300  
Washington, DC 20001

*Susan E. Shaw McBee*

SIGNATURE:

Susan E. Shaw McBee

NAME

39,294

REGISTRATION NUMBER

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Attorney Docket No.: 30098.4

In re patent application of

Kalle Nalo GmbH &amp; Co. KG

Serial No.: Unassigned

Group Art Unit: Unassigned

Filed: May 19, 2000

Examiner: Unassigned

For: SPONGE CLOTH BASED ON CELLULOSE AND PRODUCTION THEREOF

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents

Washington, D.C. 20231

Sir:

Preliminary to examination, please amend the above-identified application as follows:

**IN THE SPECIFICATION:**

Page 2, after line 14, insert --WO 97/42259 discloses sponge cloths containing reinforcing fibers and processes for producing them. The fibers can be added as additive or else originate from the cellulosic starting material if this is only incompletely dissolved. The dissolving of the cellulose material, which is pretreated with steam under high pressure, is generally effected using strongly alkaline, aqueous solutions, preferably with NaOH solutions. As well as a pore former, a blowing agent is additionally used as well at all times, for example a surfactant such as sodium lauryl sulfate.

EP-A 712 889 discloses fiber-containing cellulose/amine oxide solutions for producing mainly fibers, papers, films and food casings. Sponges are mentioned as well, but not sponge cloths.

WO 97/23552 describes cellulose sponges (not sponge cloths) and processes for producing them. These sponges are produced from a cellulose/NMMO solution which is mixed with a pore former and a blowing agent. The mixture is subsequently exposed to conditions under which the blowing agent decomposes, causing the cellulose solution to foam. This produces the large pores which are typical of sponges and which are undesirable in a sponge cloth.--

Page 8, line 17, delete "10 to 16 Torr", and insert --1.333 to 2.133 kPa (10 to 16 Torr)--;  
line 21, delete "200 Torr", and insert --26.66 kPa (200 Torr)--.

#### IN THE CLAIMS:

Please amend the following claims:

1. (Amended) A sponge cloth which is based on cellulose and has been provided with an internal reinforcement, capable of being obtained [obtainable] by [the] an amine oxide process without use of blowing agents.

Claim 3, line 1, after "claim 1", delete "or 2".

4. (Amended) The sponge cloth of [one or more of claims 1 to 3] claim 1, including at least one plasticizer.
5. (Amended) The sponge cloth of [one or more of claims 1 to 3] claim 1, impregnated with a biocidally active agent.

Claim 6, line 5, after "fibers," insert --but no blowing agent,--.

Claim 8, line 1, after "claim 6", delete "or 7".

9. (Amended) The process of claim 6, wherein the coagulation bath comprises a 5

to 50% by weight aqueous amine oxide solution[, preferably a 5 to 50% by weight aqueous NMMO solution].

Claim 11, line 1, after "claim 6", delete "or 7".

13. (Amended) The process of claim 6, wherein the pore former fraction is 30 to 90% by weight[, preferably 70 to 85% by weight,] based on the total weight of the sponge cloth raw material.
14. (Amended) The process of claim 6, wherein the cellulose fraction is 0.5 to 10.0% by weight[, preferably 1.0 to 5.0% by weight], based on the total weight of the sponge cloth raw material.

Claim 15, line 1, after "claim 6", delete "or 7".

16. (Amended) The process of claim 6 wherein the plasticizer fraction is 1 to 15% by weight[,preferably 2 to 10% by weight,] based on the dry weight of the finished sponge cloth.

Please add the following new claims:

- 17. A process according to claim 9, wherein the weight is 5 to 50% of an aqueous NMMO solution.
18. A process according to claim 13, wherein the weight is 70 to 85%.
19. A process according to claim 14, wherein the weight is 1.0 to 5.0%.
20. A process according to claim 16, wherein the weight is 2 to 10%.--

## REMARKS

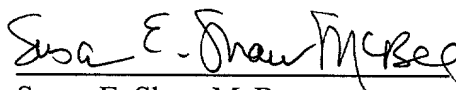
The instant amendments are being made to effect changes made to the instant §371 application during the international stage and to delete multiple dependencies of the claims and “preferably” clauses. Claim 1-20 are pending.

An early action on the merits is awaited.

Respectfully submitted,

May 19, 2000

Date



Susan E. Shaw McBee

Registration No. 39,294

Bowles Rice McDavid Graff & Love PLLC  
475 H Street, N.W.  
Suite 300  
Washington, D.C. 20001  
(202) 682 - 3711

Sponge cloth based on cellulose and production thereof

The invention relates to a cellulose-based sponge cloth which has been provided with an internal reinforcement and to a process for producing same.

- 5 The sponge cloth is highly water-absorbent and is useful in the household and industry for various cleaning and decontaminating purposes.

- Sponge cloth production by the viscose process is well known. First, pulp, especially wood pulp, is converted with sodium hydroxide and carbon disulfide into an alkaline cellulose xanthate solution known as viscose. At the same time, cotton noils are degreased with dilute, detergent-containing NaOH. The ripened viscose solution is then mixed with cotton fibers. This is generally done with the aid of a kneader. Instead of cotton fibers it is also possible to use viscose fibers as internal reinforcement. Glauber's salt (= sodium sulfate decahydrate) is then added and likewise mixed in uniformly. This sponge cloth raw material is then applied to a support, for example a foraminous endless belt, to whichever depth is desired. It is also possible to apply a mixture of viscose solution and Glauber's salt to a plastic support material, for example a net of polyethylene terephthalate (PET). The regeneration of the cellulose then takes place in a heated, alkaline coagulation bath. It can also be carried out in an acidic medium, for example dilute sulfuric acid. In the process, the internal reinforcement becomes integrated into the sponge cloth body.
- 10  
15  
20

- Glauber's salt has a very low melting point. It therefore melts in the coagulation bath and is dissolved out. The disappearing salt crystals leave pores and voids behind. Finally, the sponge cloth is washed out to rid it of salt residues and adhering reaction products. After drying, it is cut into narrow lengths, which in turn are rolled up. The roll material can then be end-itemed into cloths of the desired size.
- 25

However, the viscose process is technically very complicated and costly. In addition, appreciable efforts are needed if it is not to impact adversely on the environment. An unpleasant concomitant of the viscose process is for example the formation of foul smelling hydrogen sulfide. A sponge cloth  
5 produced by the viscose process still contains residues of  $\text{CS}_2$  or  $\text{H}_2\text{S}$ , which have to be expensively washed out. In addition, in the course of the several hours of ripening of the viscose solution, the cellulose polymer chains are more or less degraded. This leads to a pronounced broadening of the distribution curve of the degree of polymerization (DP) of the cellulose chains.  
10 Even the use of a cellulose starting material having the same degree of polymerization therefore does not consistently guarantee the same post-regeneration result. The mechanical stability of the sponge cloth will then moreover fluctuate, inter alia according to the degree of polymerization.

15 It is an object of the present invention to use an economically and ecologically better process to produce sponge cloth which contains no or at least distinctly less troublesome impurities and has a consistently high quality.

This object is achieved by a sponge cloth which is obtainable by the amine  
20 oxide process. This process utilizes solutions of cellulose in a mixture of an N-oxide of a tertiary amine and water. A particularly suitable N-oxide is N-methylmorpholine N-oxide (NMMO).

The amine oxide process is well known. Hitherto this process was used to  
25 produce especially cellulose-based fiber or film. AT-B 402 932 (Application No. 95-02100) describes the production of cellulose sponge from a solution of cellulose in a tertiary amine oxide mixed with a pore former and a blowing agent. This mixture is exposed to conditions where the blowing agent decomposes to form gaseous products and so causes a foam to form with  
30 relatively large voids. The foamed mixture is then contacted with water to precipitate the cellulose. The amine oxide can be washed out of the resulting



sponge using water. However, blowing agents are completely unsuitable in the production of sponge cloth, since they would provide neither the requisite mechanical stability nor the necessary abrasion resistance.

5 The sponge cloth of the invention can be produced from a raw material comprising cellulose dissolved in a mixture of an N-oxide of a tertiary amine and water, a pore former and a fiber reinforcement. The N-oxide is preferably N-methylmorpholine N-oxide. The raw material thus produced is then applied via a die, preferably a slot die, to a support belt. The (preferably foraminous)  
10 support belt then transports it into a spin bath. This bath comprises a 5 to 50% by weight aqueous amine oxide solution, preferably a 5 to 50% by weight aqueous NMMO solution. The spin bath causes the cellulose to precipitate and solidify. The sponge cloth web thus produced is then washed out with water and dried.

15 In a further embodiment, a material which includes cellulose dissolved in NMMO and water and a pore former is applied to a net-shaped material, for example a polyester net, from both sides. The net thus coated is then passed through spin and wash baths as described. However, an internal  
20 reinforcement is required in either case. In a cellulose sponge, in contrast, the internal reinforcement plays only a minor role in favor of the water absorption capacity.

The sponge cloth produced by the process of the invention has - under  
25 otherwise identical general conditions - a higher mechanical stability and hence better breaking strength than the sponge cloth produced by the viscose process. The reason is primarily that the inevitable polymer degradation in the ripening of the viscose solution does not occur in the process of the invention. The molecular weight distribution curve of the cellulose product of the  
30 invention is virtually identical to the distribution curve of the cellulose raw material used in the process. Since, moreover, no polymer degradation

occurs if a prolonged period elapses between the preparation of the solution and its coagulation, consistent quality is ensured. The sponge cloth of the invention is virtually free of undesirable impurities such as hydrogen sulfide and carbon disulfide. It is capable of absorbing and retaining a large amount of water. It is, moreover, generally naturally degradable and can therefore be composted without problems.

The amine oxide used in the production of the sponge cloth is virtually quantitatively recoverable and - after appropriate concentrating - reusable. It is therefore advantageous for the amine oxide in the sponge cloth raw material to be identical to the amine oxide in the spin bath and in the subsequent baths. The remaining wastewater contains hardly any residuals and it therefore has only a low chemical oxygen demand (COD).

The pore former in the process of the invention is preferably an inorganic salt which is readily water-soluble. Useful are in particular alkali metal, alkaline earth metal and ammonium salts of inorganic acids, such as chlorides (specifically NaCl and KCl, but not  $MgCl_2$ ), sulfates (specifically  $Na_2SO_4$  and  $MgSO_4$ ), phosphates (specifically  $Mg_3(PO_3)_2$ , silicates (specifically  $Mg_2[SiO_4]$  = forsterite) and carbonates (specifically  $Na_2CO_3$ ,  $CaCO_3$  and  $(NH_4)_2CO_3$ ). The diameter of the salt crystals is generally up to 2.5 mm, preferably up to 2.0 mm. But only about 1/5 of the crystals should have a diameter of less than 0.3 mm, or the sponge cloth obtained will have a reduced water absorption capacity. The pore former fraction is generally 30 to 90% by weight, preferably 70 to 85% by weight, based on the total weight of the sponge cloth raw material.

A suitable source of cellulose is pulp, especially ground wood pulp. The cellulose used in the process of the invention should have a degree of polymerization DP of 300 to 1600, preferably 400 to 600. Mixing the cellulose with the aqueous NMMO (water fraction about 40% by weight) will initially

result in the formation of a suspension. The suspension is then stirred and heated (about 70 to 120°C, preferably 85 to 95°C) under reduced pressure (preferably about 40 to 200 mbar) to remove some of the water. The water fraction decreases from about 40% by weight to about 7 to 12% by weight, and the suspension changes into a virtually homogeneous solution. The refractive index of the solution is about 1.4910 to 1.4930. The solution contains 5 to 20% by weight, preferably 10 to 15% by weight, of dissolved cellulose. The viscosity of this solution is about 1500 Pa s. When freshly prepared, the solution is only weakly alkaline (pH about 7.8). On standing, however, the NMMO decomposes into basic products which raise the pH. The preparation of the cellulose solution is described inter alia in WO 97/31970. The cellulose solution is then mixed with the other components, advantageously in a kneader. In the sponge cloth raw material thus produced, the cellulose fraction is 0.5 to 10.0% by weight, preferably 1.0 to 5.0% by weight, based on the total weight of the raw material.

The fiber reinforcement comprises natural fibers, preferably cotton, hemp, flax or viscose fibers produced by the NMMO process (lyocell). Fiber length is 5 to 50 mm, preferably 10 to 30 mm. The fibers are generally pretreated to improve the compatibility with the NMMO solution. The fiber fraction is 5 to 50% by weight, preferably 10 to 40% by weight, based on the dry weight of the sponge cloth.

Sponge cloths are relatively brittle in the dry state. To remedy this disadvantage, plasticizing substances may be added to the sponge cloth raw material. Particularly useful plasticizers are washed out only very slowly, if at all, and therefore retain their effectiveness ideally over the entire use period. Useful plasticizers are in particular starch and starch derivatives, low molecular weight cellulose derivatives, polyvinyl alcohol, vinylpyrrolidone/vinyl alcohol copolymers, polyvinyl acetate, vinyl methyl ether/maleic anhydride copolymers (@Gantrez), urea, hexamethylenediamine, carrageenan, alginates

- (especially sodium alginate), guar and guar derivatives [especially cationic guar derivatives (@Meyprofloc), carboxymethylhydroxypropyl guar (@Jaguar CMHP) and highly substituted carboxymethyl guar (@Jaguar CMHS)], galactomannan, xanthan gum, polysaccharides with side chains (@Rhodigel),
- 5 heteropolysaccharides (@Rhodicare), alkylpolyethoxyammonium lactate (@Genamin), polyester interpolymers (@Cassapret), phosphoric esters (@Hostaphat), condensation products based on saturated or unsaturated fatty acids of various chain lengths and taurine, methyltaurine or hydroxyethane-sulfonic acid, N-cocoylglutamic acid monosodium salt (@Hostapon), fatty
- 10 alcohol polyglycol ether (@Genapol), phthalic diesters or quaternary nitrogen compounds (@Leomin). The plasticizer fraction is 1 to 15% by weight, preferably 2 to 10% by weight, based on the dry weight of the finished sponge cloth.
- 15 The sponge cloth of the invention may finally also be impregnated with biocidally - especially fungicidally, bacteriocidally and/or algicidally - active substances (cf. DE-U 296 18 058). A biocidal finish is particularly sensible when moistened sponge cloths are likely to remain for a prolonged period inside a polymeric film pack. Particularly preferred biocides are isothiazolone,
- 20 benzisothiazolone and benzimidazole derivatives. Fungicidal or bacteriocidal activity is also exhibited by cationic, surface-active ammonium salts having long-chain, saturated or unsaturated ( $C_6$ - $C_{24}$ )alkyl groups, especially ( $C_8$ - $C_{18}$ )alkyltrimethylammonium chlorides, di( $C_{10}$ - $C_{18}$ )alkyldimethylammonium chlorides or alkylbenzyldimethylammonium chlorides. As well as
- 25 the chlorides of tertiary ammonium compounds, however, bromides, acetates, propionates, sorbates, benzoates or sulfates may also be used. Such compounds are disclosed in EP-A 286 009 for example. Bacteriocidal activity is finally also exhibited by dipyrindyl sulfide and its bis-N-oxide or 1-alkyl- and 1-alkenylpyridinium salts (for example 1-laurylpyridinium chloride). Similarly,
- 30 aluminosilicates or zeolites (@Zeomic from Shinanen Zeomic Co. Ltd., Japan, CAS No. 1318-02-1) in which silver or copper has been incorporated exhibit

a biocidal effect. The same effect can also be achieved by impregnating with glycerol or propylene glycol.

5 Finally, the sponge cloths of the invention may also be moistened. A particularly useful moistener is a 2 to 8% by weight magnesium chloride solution which may additionally include 0.2% by weight of a scent, for example of an oil having a citrus, pine or similar scent, in emulsified form.

10 The invention also provides the process for producing a sponge cloth which is based on cellulose and has an internal reinforcement, which comprises the steps of:

- 15 (a) providing a mixture which includes cellulose dissolved in the N-oxide of a tertiary amine and water and also at least one pore former and fibers,
- (b) spreading the mixture onto a transportation belt,
- (c) treating the layer with a coagulation bath comprising a dilute aqueous amine oxide solution to dissolve out the pore former,
- (d) washing the remaining amine oxide out,
- 20 (e) drying the sponge cloth web and
- (f) end-iteming it.

25 The sponge cloth raw material is advantageously produced by mixing the individual ingredients in a kneader. It is advantageous to mix in the pore former and any color pigment last. The spreading of the raw material on the transportation belt may be effected for example with the aid of a slot die.

30 By end-iteming a person skilled in the art will understand primarily the operation of cutting the moist or dry sponge cloth length to the desired size and packing with or without printing.

In one variant of the process according to the invention, steps (a) and (b) are modified. Step (a) dispenses with the fibers; instead, step (b) comprises applying the mixture via a die in a thin layer to both sides of a plastic net.

- 5 The examples hereinbelow illustrate the invention. Percentages are by weight, unless otherwise stated.

Example 1:

510 g of ground wood pulp (@Cellunier F from Rayonier) having an average  
10 degree of polymerization of 535 (determined by the cuoxam method) was steeped up in 5087 g of a 60% NMMO solution. The pH of the steep was then adjusted to 11 with NaOH. The steep was then stirred and heated under reduced pressure with increasing temperature to distill off water until the NMMO content was 87.7%, based on the total weight of water and NMMO,  
15 and the monohydrate was present (discernible from a refractive index of 1.4820). During this phase, which lasted about 4 hours, the vacuum was maintained at 10 to 16 Torr. After a further 2 to 3 hours of stirring at about 85 to 95°C, all the pulp had gone into solution.

20 In order that less water may evaporate, the vacuum was adjusted to about 200 Torr during this period. The refractive index then settled down at about 1.4910 to 1.4930, which corresponds to a water content of 7.5 to 9%.

The solution thus prepared was admixed with 850 g of cotton wetted with an  
25 appropriately heated solution consisting of caustic soda and a degreaser and kneaded. In a second kneading process, the pulp-cotton solution was admixed with 30 kg of sodium chloride pore former and 3 g of a blue pigment while maintaining a temperature ensuring the requisite fluidity.

The sponge cloth obtained had the following properties:

5

10

15

Properties	Unit	Value	Standards
Basis weight	g/m <sup>2</sup>	263	DIN 53 854 (absolutely dry)
Thickness in moist state	mm	5.3	DIN 53 855, 0.02 N/cm <sup>2</sup>
Thickness in dry state	mm	2.4	DIN 53 855, 0.2 N/cm <sup>2</sup>
Water absorption capacity	l/m <sup>2</sup>	5.4	
Veslic test	Rub cycles	98	
Abrasion	g/m <sup>2</sup>	41	DIN 53 528
Wet ultimate tensile stress strength	N/15 mm	25	DIN EN ISO 527-1
Wet ultimate tensile stress extension	%	19	DIN EN ISO 527-1

Example 2

Example 1 was repeated except that the amount of the pore former (NaCl) was reduced to 25 kg. The sponge cloth obtained had the following properties:

5

Properties	Unit	Value	Standards
Basis weight	g/m <sup>2</sup>	380	DIN 53 854 (absolutely dry)
Thickness in moist state	mm	5.9	DIN 53 855, 0.02 N/cm <sup>2</sup>
Thickness in dry state	mm	2.8	DIN 53 855, 0.2 N/cm <sup>2</sup>
Water absorption capacity	l/m <sup>2</sup>	6.3	
Veslic test	Rub cycles	112	
Abrasion	g/m <sup>2</sup>	38	DIN 53 528
Wet ultimate tensile stress strength	N/15 mm	30	DIN EN ISO 527-1
Wet ultimate tensile stress extension	%	25	DIN EN ISO 527-1

10

15



What is claimed is:

1. A sponge cloth which is based on cellulose and has been provided with an internal reinforcement, obtainable by the amine oxide process.
- 5 2. The sponge cloth of claim 1, wherein the amine oxide used in the process is N-methylmorpholine N-oxide.
3. The sponge cloth of claim 1 or 2, wherein the internal reinforcement comprises cotton fibers, viscose fibers or a polymeric net.
- 10 4. The sponge cloth of one or more of claims 1 to 3, including at least one plasticizer.
- 15 5. The sponge cloth of one or more of claims 1 to 3, impregnated with a biocidally active agent.
6. A process for producing a sponge cloth which is based on cellulose and has been provided with an internal reinforcement, which comprises
  - 20 (a) providing a mixture which includes cellulose dissolved in the N-oxide of a tertiary amine and water and also at least one pore former and fibers,
  - (b) spreading the mixture onto a transportation belt,
  - 25 (c) passing the layer through a coagulation bath comprising a dilute aqueous amine oxide solution to dissolve out the pore former,
  - (d) washing the remaining amine oxide out,
  - (e) drying the sponge cloth web and
  - (f) end-iteming it.
- 30

7. A process for producing a sponge cloth which is based on cellulose and has an internal reinforcement, which comprises
- 5 (a) providing a mixture which includes cellulose dissolved in the N-oxide of a tertiary amine and water and also at least one pore former,
- (b) applying the mixture to both sides of a polymeric net,
- (c) passing the layer through a coagulation bath comprising a dilute aqueous amine oxide solution to dissolve out the pore former,
- 10 (d) washing the remaining amine oxide out,
- (e) drying the sponge cloth layer and
- (f) end-iteming it.
8. The process of claim 6 or 7, wherein the N-oxide of a tertiary amine is N-methylmorpholine N-oxide.
- 15 9. The process of claim 6 or 7, wherein the coagulation bath comprises a 5 to 50% by weight aqueous amine oxide solution, preferably a 5 to 50% by weight aqueous NMMO solution.
- 20 10. The process of claim 6, wherein the fibers fraction is 5 to 50% by weight, preferably 10 to 40% by weight, based on the dry weight of the sponge cloth.
- 25 11. The process of claim 6 or 7, wherein the pore former is an alkali metal, alkaline earth metal or ammonium salt of an inorganic acid.
12. The process of claim 11, wherein the pore former is sodium sulfate or magnesium sulfate.

13. The process of claim 6 or 7, wherein the pore former fraction is 30 to 90% by weight, preferably 70 to 85% by weight, based on the total weight of the sponge cloth raw material.
- 5 14. The process of claim 6 or 7, wherein the cellulose fraction is 0.5 to 10.0% by weight, preferably 1.0 to 5.0% by weight, based on the total weight of the sponge cloth raw material.
- 10 15. The process of claim 6 or 7, wherein the mixture additionally includes at least one plasticizer and/or at least one biocidally active agent.
- 15 16. The process of claim 6 or 7, wherein the plasticizer fraction is 1 to 15% by weight, preferably 2 to 10% by weight, based on the dry weight of the finished sponge cloth.

Abstract:

The invention relates to a sponge cloth based on cellulose and having an internal reinforcement, obtainable by the amine oxide process. In the process, a solution of cellulose in an aqueous amine oxide is first produced, which is then mixed with at least one pore former and fibers. The mixture is spread on a conveyor belt which is then guided through a coagulating bath comprised of a diluted aqueous amine oxide solution whose temperature is high enough such that the pore former melts and is dissolved out. Remaining amine oxide is washed out. After subsequent drying the sponge cloth layer is end-itemed. Contrary to the viscose process polymer degradation does not occur in the inventive process which results in a sponge cloth having an improved mechanical stability. A plastic net can replace the fiber reinforcement. The process is especially ecological and economical. The sponge cloth is highly water-absorbent and can be used for household or industrial purposes, especially for cleaning and decontaminating purposes.

**DECLARATION AND POWER OF ATTORNEY**

As a below named inventor, I HEREBY DECLARE:

THAT my residence, post office address, and citizenship are as stated below next to my name:

THAT I believe I am the original, first, and sole inventor (if only one inventor is named below) or an original, first, and joint inventor (if plural inventors are named below or in an attached Declaration) of the subject matter which is claimed and for which a patent is sought on the invention entitled

**SPONGE CLOTH BASED ON CELLULOSE AND PRODUCTION THEREOF**

the specification of which (check one)

\_\_\_\_\_ is attached hereto.

XX was filed on November 25, 1998 as United States Application Number or PCT International Application Number PCT/EP98/07599 and was amended on \_\_\_\_\_ (if applicable).

THAT I do not know and do not believe that the same invention was ever known or used by others in the United States of America, or was patented or described in any printed publication in any country, before I (we) invented it;

THAT I do not know and do not believe that the same invention was patented or described in any printed publication in any country, or in public use or on sale in the United States of America, for more than one year prior to the filing date of this United States application;

THAT I do not know and do not believe was first patented or made the subject of an inventor's certificate that issued in any country foreign to the United States of America before the filing date of this United States application if the foreign application was filed by me (us), or by my (our) legal representatives or assigns, more than twelve months (six months for design patents) prior to the filing date of this United States application;

THAT I have reviewed and understand that contents of the above-identified specification, including the claim(s), as amended by any amendment specifically referred to above;

THAT I believe that the above-identified specification contains a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the invention, and sets forth the best mode contemplated by me of carrying out the invention; and

THAT I acknowledge the duty to disclose to the U.S. Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I HEREBY CLAIM foreign priority benefits under Title 35, United States Code §119(a)-(d) of §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application

which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number	Country	Foreign Filing Date	Priority Claim?	Certified Copy Attached?
197 53 546.1	GERMANY	December 3, 1997	YES	YES

I HEREBY CLAIM the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

U.S. Provisional Application Number	Filing Date

I HEREBY CLAIM the benefit under Title 35, United States Code, §120 of any United States application(s), or §365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

U.S. Patent Application Number	PCT Patent Application Number	Patent Filing Date	Parent Patent Number

I HEREBY APPOINT the following registered attorneys and agents of the law firm of Bowles Rice McDavid Graff & Love PLLC to have full power to prosecute this application and any continuations, divisions, reissues, and reexaminations thereof, to receive the patent, and to transact all business in the United States Patent and Trademark Office connected therewith:

(2)	Susan E. Shaw McBee	Reg. No.	39,294
	Matthew Mulkeen	Reg. No.	44,250

and I request that all correspondence be directed to:

Susan E. Shaw McBee  
Bowles Rice McDavid Graff & Love PLLC  
475 H Street, N.W., Suite 300  
Washington, D.C. 20001  
(202) 682-3711  
(202) 682-3087

I UNDERSTAND AND AGREE THAT the foregoing attorneys and agents appointed by me to prosecute this application do not personally represent me or my legal interests, but instead represent the interests of the legal owner(s) of the invention described in this application.

I FURTHER DECLARE THAT all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so make are punishable by a fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

1-00  
Name of First Inventor Leo Mans  
Residence An der Hasenquelle 8, D-55120 Mainz, Federal Republic of Germany  
Citizenship German DEX  
Post Office Address An der Hasenquelle 8, D-55120 Mainz, Federal Republic of Germany  
Inventor's signature Leo Mans  
Date April 10, 2000

2-00  
Name of Second Inventor Klaus-Dieter Hammer, Ph.D.  
Residence An der Hasenquelle 25, D-55120 Mainz, Federal Republic of Germany  
Citizenship German DEX  
Post Office Address An der Hasenquelle 25, D-55120 Mainz, Federal Republic of Germany  
Inventor's signature Klaus-Dieter Hammer  
Date April 10, 2000